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Quality Assurance of Fats and Oils

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1. INTRODUCTION

The quality of fats and oils is dictated by several physical and chemical parameters that are dependent on the source of oil; geographic, climatic, and agronomic variables of growth in the case of plant oils as well as processing and storage conditions. Thus, quality assurance criteria may depend partly on the type of oil under investigation as well as on other factors that may vary depending on the intended use and regulations that vary from country to country (1-3).

Edible oils may originate from animals both land-based and aquatic, higher plants, and algal sources. Regardless of the source, the extraneous matters such as large pieces of wood, metal pieces, soil, and so one should be eliminated. For oilseeds, these are usually passed through a magnetized sieve. However, this process does not eliminate environmental pollutants that might exist endogenously or have been introduced into the raw material. The physical state of food lipids, mainly their crystallinity and whether they exist in the liquid or solid form, is dictated primarily by the degree of saturation/unsaturation of the oil. As a result,

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the approximate composition of the source material must be determined. For example, solid fat content may be estimated by low-resolution nuclear magnetic resonance (NMR) spectroscopy. Near-infrared (NIR) spectroscopy allows determination of fat content and other components of oilseeds. The color of the oil, which is dependent on several factors, may be determined visually or with a Lovibond tintometer or other handheld color measuring devices (4). The color may be from carotenoids, chlorophylls, or other components. When the harvested seeds are immature, often chlorophyll content of the resultant oil is high and this affects the stability of products (5). Chlorophylls are photosensitizers; hence, their presence leads to enhanced photo-oxidation of the oil. Obviously, agronomic conditions, season of the harvest, and many other related factors affect the quality of the resultant oil. In this regard, soil may affect the content of certain unwanted minerals, such as cadmium, in the seeds, but these do not usually end up in the oil. Furthermore, as explained earlier, other contaminants may be introduced into the raw material during harvest, processing, and transport. Thus, fats and oils and their source material have to be tested for the presence and level of contaminants. In addition, diseases and pests always lead to decreased quality of oils, as reflected in their high acid values (6). In the case of animal fat, every effort should be made to process the raw material in the fresh state or use fresh-frozen material to ensure premium quality of the product.

In the extraction of edible oil from seeds, cleaning and subsequent conditioning of the seeds followed by heating during or immediately after crushing are needed as these deactivate the endogenous enzymes and help in releasing of the oil. After expelling part the oil, the resultant leftover material may be flaked and then extracted with hexanes. The expelled and solvent-extracted oils are then combined and desolventized to afford crude oil.

The edible oils, after rendering or extraction from source material, may be subjected to degumming, refining, bleaching, deodorization, and possibly winterization and blending and hydrogenation and/or addition of stabilizers/antioxidants. Discussion of these steps in any detail is beyond the scope of this overview. However, each processing step carries with it many advantages and some disadvantages. To explain these briefly, it is essential to first examine the constituents of fats and oils in a cursory manner.

Edible oils are composed of triacylglycerols (triglycerides) as their main components. The phospholipids are minor components that are generally removed during the degumming process (7). The recovered phospholipids, often called lecithin, may possibly be dietary supplements. Free fatty acids are then eliminated during the refining process, and bleaching of the oil leads to the removal of colored materials as well as decomposition of hydroperoxides to secondary oxidation products. The deodorization step is then designed to remove the odorous secondary oxidation products from the oil. However, many useful minor components present in the oils are also removed during the deodorization process. The deodorizer-distillate is often high in the content of tocopherols and tocotrienols that can be removed, purified, and sold as dietary supplements or used in specialty applications. The final oil after refining, bleaching, and deodorizing (RBD) may further be subjected to

winterization, a cooling process that allows the removal of more saturated fats as well as possible blending. Some oils may also be subjected to hydrogenation to enhance their oxidative stability. However, hydrogenation often leads to the production of 30–50% *trans*-fats that are a health concern because of their potential harmful effect on the cardiovascular system (8). Therefore, novel formulations with more saturated oils in the mix has become popular (9).

Among the parameters often checked or evaluated for quality assurance of edible oils are those related to the makeup of the oil or their properties. Table 1 summarizes a list of parameters usually employed to assess quality of edible fats and oil. However, not all parameters listed may be evaluated for each oil.

In addition to parameters listed in Table 1 that dictate the quality of fats and oils, storage and transport conditions are of considerable importance as they determine the final quality of the oil. Obviously, of the above factors, fatty acid composition and oxidative stability are of utmost importance, both from nutritional and sensory quality viewpoints. In general, intake of omega-3 fatty acids in the western world is much less than desired. Nutritionally, one would like to have a ratio of 1:2–1:5 for omega-3 to omega-6 fatty acids in the diet. However, a high content of omega-3 fatty acids in edible oils is responsible for their rapid quality deterioration. Hence, much effort has been made to eliminate the omega-3 fatty acids, mainly linolenic acid, from vegetable oils. However, recent trends have reflected the concern about low intake of omega-3 fatty acids and its deleterious effects.

Adulteration of fats and oils is another matter of concern, which might occur accidentally or deliberately. Rendering of pork fat and beef tallow in the same equipment without proper washing is an example of accidental and unintended contamination/adulturation. However, often cheaper oils have been sold in place of, or mixed with, more expressive oils. Thus, before to the recognition of health benefits of hazelnut oil, this oil was an adulterant in olive oil (10). As mentioned earlier, different oils have considerably different sterol compositions. Thus, sterols could be a means of identifying adulterants because often fatty acid compositions of the adulterant and the original oils are similar (11-13).

In addition, depending on the intended use, the quality of oil during storage and use must be monitored. The oils may undergo hydrolytic rancidity, autoxidation, photo-oxidation, and thermal oxidation. The latter type of oxidation is observed primarily in the frying oil and causes quality deterioration that must be monitored with different parameters such as color, viscosity, polar components and polymers, among others (14,15). Obviously, oils that are highly unsaturated are not suitable for frying purposes. On the contrary, autoxidation is a process that proceeds slowly for properly stored oils. However, if the oil is kept in clear bottles, photo-oxidation may occur, especially when photosensitizer chlorophyll is present. Thus, parameters of interest for quality assurance of fats and oils begin at the farm gate and continue up to the dinner table, which includes proper holding and use of oil at home after purchase that, despite its importance, is often ignored by most consumers.

The following sections provide some further details about determination of quality of fats and oils. Other specifics may be found in several chapters in this series and in several other publications.

TABLE 1. Quality Parameters of Fats and Oils.

Parameter	Details
Fatty acid composition and distribution	Percentage of total; depends on the type of material
Relative density	At 20°C or 40°C relative to water at 20°C (<1)
Refractive index	At 40°C
Viscosity	At 20°C
Color	Visual, Lovibond or Colormet
Turbidity	Visual or instrumental
Solidification point, titer, solid fat content, and cooling curve	For water-insoluble fatty acids
Odor and taste	Sensory evaluation
Saponification value	mg KOH/g
Iodine value (IV)	g iodine/100-g sample (WIJS method)
Unsaponifiable matter	g/kg
Acid value (AV)	mg KOH/g
Smoke, flash and fire points	°C
Oxidative state	
Peroxide value (PV)	meq oxygen/100-g sample
Thiobarbituric acid reactive substances (TBARS)	μmol/g
para-Anisidine value (p-Anv)	mg/kg
TOTOX	2PV + *p*-AnV
OSI, Rancimat and AOM value	—
Polar Lipids	Percentage
Polymers	Percentage
Volatile mater (%)	At 105°C
Phosphorus	mg/kg
Iron, copper, lead, arsenic	mg/kg
Cadmium	μg/kg
Trans-fatty acids	Percentage; measured at ~10 μ
Cholesterol content	Percentage, mainly for animal fat
Contaminants and foreign matter, including plasticizers (%)	—
Carotenoids and chlorophylls	mg/kg
Squalene	$C_{30}H_{50}$
Sterols	GC determination
Tocols	HPLC determination
Synthetic antioxidants	BHA, BHT, TBHQ, PG
Antifoaming agents	Dimethyl polysiloxane, singly or with silicon dioxide
Metal chelators	Citric acid or citrates, phosphoric acid
Crystallization inhibitor	Oxystearin
Adulterants	Fingerprinting using sterols or other minor components

Abbreviations: BHA, butylated hydroxyanisole; BHT, butylated hydroxytoluene; TBHQ, *tert*-butylhydroquinone; and PG, propyl gallate.

2. OIL COMPOSITION

Fats and oils contain various classes of compounds (16). These componds are primarily neutral lipids that include triacylglycerols (triglycerides) with lower amounts of diacylglycerols (diglycerides), monoacylglycerols (monoglycerides),

and free fatty acids. Partial acylglycerols are produced by hydrolysis of triacylglycerols. Some oils such as cottonseed oil contain about 10% diacylglycerols. The amount of free fatty acids should be less than 0.1%, preferably less than 0.05% in freshly refined oils. In addition, polar lipids, mainly phospholipids, and to a lesser extent, glycolipids are present. The content of phosphorus in crude oils may reach 500 ppm, and in refined oils, which from phospholipids, the content is generally less than 5 ppm, and may be below 2 ppm. In addition, fats and oils, in general, contain a small amount of unsaponifiable matter, generally at 0.3–2.0% mainly tocopherols, tocotienols, phytosterols, hydrocarbons (e.g., squalene and carotenes), among others. Phenolics such as hydroxytyrosol and oleuropein might also be present (17). Trace metals, mainly iron and copper, and other components often exist. The content of iron and copper in freshly refined oils should be less than 0.1 and 0.01 ppm, respectively. Crude palm oil was 0.6 ppm for iron, 6.05 ppm for copper, 0.6 ppm for magnesium, 1.2 ppm for chromium, and 2.2 ppm for nickel (18).

The triacylglycerols of fats and oils contain a range of fatty acids, and their arrangements on the glycerol backbone may vary, depending on the source material. High-performance liquid chromatography as well as gas liquid chromatography may be used for separation and tentative identification of individual triacylglycerols based on their carbon number.

In neutral oils and fats, the fatty acids are not usually randomly distributed among different positions on the glycerol backbone and are associated in particular patterns. As an example, saturated fatty acids such as palmitic and stearic acids are associated with the *sn*-1 and *sn*-3 positions of soybean oil, albeit at higher proportions in the *sn*-1 position. However, the reverse is observed at high content of saturated fatty acids. Linoleic acid is preferably in the *sn*-2 position, whereas oleic acid is randomly distributed among the three positions. Linolenic acid is primarily at *sn*-2 followed by *sn*-1 and *sn*-3 positions. The stereospecific distribution of fatty acids has a marked effect on the oxidative stability of the resultant oils, and their presence at the *sn*-2 position helps their stability (19).

The fatty acids present in fats and oils may be analyzed after their hydrolysis and subsequent conversion by methylation to volatile methyl esters. In this Process, different methylating agents may be used, and these are methanol/sulfuric acid (20) or methanol-BF_3 (21). The methyl esters so produced are then identified with gas chromatography. Standard fatty acids methyl esters are often used for tentative identification purposes. For determination of fatty acid isomers, including *trans*-fatty acids, it is necessary to use appropriate columns and conditions for analysis.

Other parameters that are indirectly related to the composition of edible oils include iodine value and saponification value. The iodine value is a simple chemical constant for a fat or oil. It measures unsaturated or the average number of double bonds in fats and oils. Iodine value is defined as the number of grams of iodine that could be added to 100 g of oil, which is measured with the AOCS Method cd 1-25 (22). Meanwhile, saponification value is a measure of the alkali-reactive groups in fats and oils and is defined as the mg of KOH needed to saponify 1 g of oil. Shorter chain fatty acids give higher saponification values than do longer chain fatty acids.

3. MINOR COMPONENTS

Polar lipids. Polar lipids, mainly phospholipids, are present in fats and oils, and these originate primary as components of cell membranes and serve biological functions in the cells. Among phospholipids present are phosphotidylcholine (PC), phosphatidylethanolamine (PE), and phosphatidylinositol (PI). In general, saturated fatty acids are present at the *sn*-1 and unsaturated fatty acids at the *sn*-2 positions of phospholipid molecules.

Sphinogolipids are also important bioactive components of all membranes. Their hydrolysis products participate in regulation of growth, differentiation, and apoptosis by cells. They may also participate in reducing the cancer risk in humans; colon and skin cancers are particularly inhibited.

The content of polar lipids is reduced during oil refining. Degumming removes most polar lipids. However, refining, bleaching, and deodorization would also bring about a reduction in the content of polar lipids.

4. UNSAPONFIABLES MATTER

In general, unsaponifiable matters are resent in edible oils at less than 2% (23,24), which include tocopherols/tocotrienols, other phenolics, phytosterols, hydrocarbons, among others. The content of these unsaponifiable matters is varied in different oils and depending on the extent of oil refining. Although tocols and other phenolics as well as phytosterols are removed during different stages of oil refining, their main reduction occurs during deodorization of oils. Thus, deodorizer distillates rich in tocols and sterols may be used for production of these components, which may ultimately be used as nutraceuticals or for other food applications.

The dominance of tocopherols, namely, alpha-, beta-, gamma-, and delta- and the corresponding tocotrienols, depends on the type of oil under investigation. Thus, tocotrienols occur primarily in palm and rice bran oils. Meanwhile, tocopherols are more widely present in different oils. However, their proportions in different oils is dependent on the source material. As an example, sunflower oil contains mainly alpha-tocopherol and very small amounts of other tocopherols, whereas soybean oil contains mainly gamma-tocopherol with decreasing amounts of delta-, alpha-, and beta-tocopherols as determined by high-performance liquid chromatography.

Another group of unsaponifiable matter is phytosterols, fatty acid esters of phytosterols, and sterol glycosides. Again, their amount is reduced during processing. The presence of high amounts of phytosterols in soybean germ oil has been documented, which include beta-sitosterol, campesterol, stigmasterol, and Δ5-avenasterol. Phytosterols are recognized for their cholesterol-lowering properties (25). Phytosterols are usually analyzed with gas chromatography.

The hydrocarbons present in oils are composed mainly of squalene and carotenoids such as beta-carotene, among other carotenes. In addition, oxygenated

derivatives of carotenoids may be present. Palm oil serves as a rich source of carotenoids at 500–700 ppm.

5. CHARACTERISTICS OF FATS AND OILS

Fats and oils pass through a series at crystallization phases at cooling (26). Therefore, when melting such crystals, the melting points of fats and oils provides an estimate of their degree of saturation/unsaturation that parallels the saturation/unsaturation pattern dictated by their fatty acid constituents. *Trans*-fats, when present, have a higher melting point than do their *cis*-counterparts because of better packing of *trans*-fatty acids when compared with their *cis* counterparts. The melting behavior and crystal structures are major factors that are important when using such products in different applications such as in confectionary products.

Fats and oils often show multiple melting points. As an example, tristearin has three melting points at 52°C, 64°C and 70°C, because fats and oils solidify in more than one crystal form; this property is known as polymorphism. Crystallization of fats and oils occurrs in two stages of nucleation and growth.

Titer is another variable often recorded for fats and oils (16). It measures the solidification point of the fatty acids as per AOCS Method ce 12-59 (22).

The density of liquid oils is dependent on their fatty acid composition, minor components, and temperature. An equation taking these into account was developed by Pantzaris (27) using iodine value, saponification value, and temperature. The density of liquid oils is in the range of 0.909–0.921 and for solid fats varies between 0.858 and 0.893. The lower values are for more solid fats such as lard and tallow. In a similar way, the viscosity of various vegetable oils depends on their fatty acids. Generalized methods have been developed that allow calculation of density and viscosity of different oils. Coupland and McClements (28) and Fisher (29) have related viscosity and density, refraction, surface tension, and other physical properties. Viscosity of fats and oils also depends on the temperature.

The refractive index of oils depends on their molecular weight, fatty acid chain length, degree of unsaturation, and degree of conjugation. Triacylglycerols have higher refractive indices than do their constituent free acids. Values of refractive index for different oils generally vary between 1.447 and 1.482.

Smoke point is another characteristic that is important if oils are with for frying. The temperature at which smoking is observed with actual frying or heating is measured with AOCS Method Ca 9a-48 (22). Smoke point depends primarily on the content of free fatty acids as they are more volatile than their corresponding triacylglycerols.

6. COLOR AND APPEARANCE

Most oils are yellow-red or amber liquids. The color is from the presence of chlorophylls and carotenoids. The colored bodies are often removed during the bleaching

process. Often lighter color has been associated with better quality oils, especially for salad oils and shortenings.

The presence of chlorophylls not only renders a green color to products, but also they act as sensitizers for fats and oils oxidation. However, unrefined olive oils contain 1–20 ppm of chlorophylls that are considered important as extra virgin quality indicators for this oil.

Carotenoids are present in edible oils at different levels. These are powerful antioxidants against both autoxidation and photo-oxidation. Therefore, attempts have been made to retain them or recover them, as in the case of palm oil. However, carotenoids may be degraded to colorless products at high temperatures exceeding 150°C.

The color of edible oils is measured by the so-called Wesson method that is described in the AOCS Method Ce 136-45 (22) by comparison with red and yellow Lovibond glasses of known characteristics. The oil is placed in Lovibond containers that are 1 or 5.25 inches, and the color superimposes a mixture of red and yellow standards to adjust to the color of the sample. Although color is three-dimensional, the brightness factor is not considered. Yellow is needed to allow the color to look similar, but yellow is considered unimportant in this method and only the redness is measured. This method is the one used by the U.S. edible oil industry. The British standard, however, uses Lovibond tintometer. The geometry and color scales for these two methods are different, as in the tintometric method, a series of permanently colored glass standards of red, yellow, and blue are used. Each standard color is numbered. The addition of the blue color field provides a greater degree of brightness and greenness than in the Wesson method (30).

7. OXIDATIVE QUALITY AND STABILITY TESTS

Oxidative stability of edible oils depends primarily on their fatty acid composition and, to a lesser extent, in the stereospecific distribution of fatty acids in the triacylglycerol molecules. The presence of minor components in the oils also affects their oxidative stability. A detailed discussion of oxidative processes in fats and oils is provided elsewhere in this series. Oxidation may occur via different routes and includes autoxidation, photo-oxidation, thermal oxidation, and hydrolytic processes, all of which lead to production of undesirable flavor and products harmful to health. Flavor and odor defects may be detected by sensory analysis or by chemical and instrumental methods. However, chemical and instrumental procedures are often employed in the processing and during usage of edible oils. Indicators of oxidation are those that measure the primary or secondary products of oxidation as well as those from hydrolytic processes or from thermal oxidation, including polymers and polar components (15).

Peroxide value. Peroxide value (PV) is the most common measurement of lipid oxidation. Hydroperoxides have no flavor or odor of their own, but they are unstable and break down rapidly to other products such as aldehydes that have a strong, disagreeable flavor and odor. Peroxide value measures the miliequivalents of oxygen

(hydroperoxides) per gram of oil. The iodometric AOCS Method Cd 8-53 (22) is used. PV is most widely used for determination of edible oil quality. The maximum PV of 0.1 and preferably less than 0.05 is expected for freshly refined oils. A peroxide value of higher than 10 meq/kg is considered unacceptable. Conjugated dienes and trienes absorbing at 234 and 268 nm, respectively, are directly related to hydroperoxides and are often used in addition or in place of PV.

8. CARBONYL COMPOUNDS

Carbonyl compounds in oxidized fats and oils are the secondary oxidation products that originate from decomposition of hydroperoxides. They usually have low threshold values and hence are responsible for off-flavor development in oxidized oils. Therefore, content of carbonyl compounds corresponds with sensory data.

Anisidine value. The *p*-anisidine value (*p*-AnV) measures the amount of unsaturated aldehydes in fats and oils. In this method, *p*-anisidine reacts with aldehydes in acetic acid to afford a yellowish color that is measured at 350 nm. The color intensity depends on the amount of aldehydes as well as on their structure. The AOCS Method Cd 18-90 (22) has been standardized for anisidine value analysis. The Totox value, which is 2 PV + *p*-AnV, provides information about the current status of oxidation as well as its history and is used by the industry.

Thiobarbituric Acid Value. The 2-thiobarbituric acid (TBA) test is a popular method for measuring sensory oxidation products. It is based on the formation of a colored complex between two molecules of TBA reagent with one molecule of malonaldehyde or TBA reactive substances (TBARS). This intensity of the pink chromogram is measured at 532 nm.

Gas Chromatographic Methods. Gas chromatographic methods may be used for measuring volatile oxidation products. Static headspace, dynamic headspace, or direct injection methods may be employed. Specific aldehydes may be measured as indicators for oxidative stability of oils and fats. Thus, propanal is an and as indicator for stability of omega-3 fatty acids, whereas hexanal is best for following the oxidative stability of omega-6 fatty acids.

Free Fatty Acid/Acid Value. Hydrolytic processes lead to the formation of free fatty acids by splitting of acylglycerols that can affect flavor. The Standard AOCS Method Ca 5a-40 and Cd 3a-63 (22) for acid value are commonplace. Free fatty acids are normally calculated as free oleic acids on a percentage bases. Free fatty acids are important quality indicators during processing and storage of fats and oils. They are also found during frying of fats and oils. The amount of moisture from foods fried and the frying temperature are important.

9. POLYMERS AND POLAR COMPONENTS

The content of polymers and polar components in oils increases during frying process. Size exclusion chromatography and HPLC may be used for the analysis of such components. The content of polar lipids should not exceed about 20%.

10. ANTIOXIDANTS

Antioxidants are used widely in fats and oils products to delay oxidative processes. Synthetic antioxidants, namely, butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), *tert*-butylhydroquinone (TBHQ), and propyl gallate (PG), are permitted antioxidants that are frequently used in products. Their presence and concentration may be determined with HPLC and GC methods. Meanwhile, metal chelators such as citric acid may be determined by HPLC analysis.

11. ADULTERATION

Adulteration of fats and oils is an old problem. Many older tests involved determination of physical properties such as refractive index, melting point, and viscosity. However, color tests were later used for this purpose. Thus, Baudonin reaction for sesame oil and the Halpben test for cottonseed oil have been noted. In both cases, a compound characteristic to an oil determines the presence of the oil. However, today such detections and quantitations are carried out with GC and HPLC procedures. Thus, cholesterol and phytosterols may be determined by gas chromatography for fingerprinting purposes; however, fatty acid analysis might also be used for higher levels of contamination (31). Detailed discussion of issues related to oil authentication and adulteration has taken place (11).

12. POLLUTANTS

Environmental pollutants such as pesticides and herbicides may be present in fats and oils. In this connection, special attention should also be paid to the presence of polychlorinated biphenyls (PCB) as well as dioxin as well as polycyclic aromatic hydrocarbon in the oil. The presence of high levels of such unwanted matters in the oil may render them unfit for edible purposes.

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